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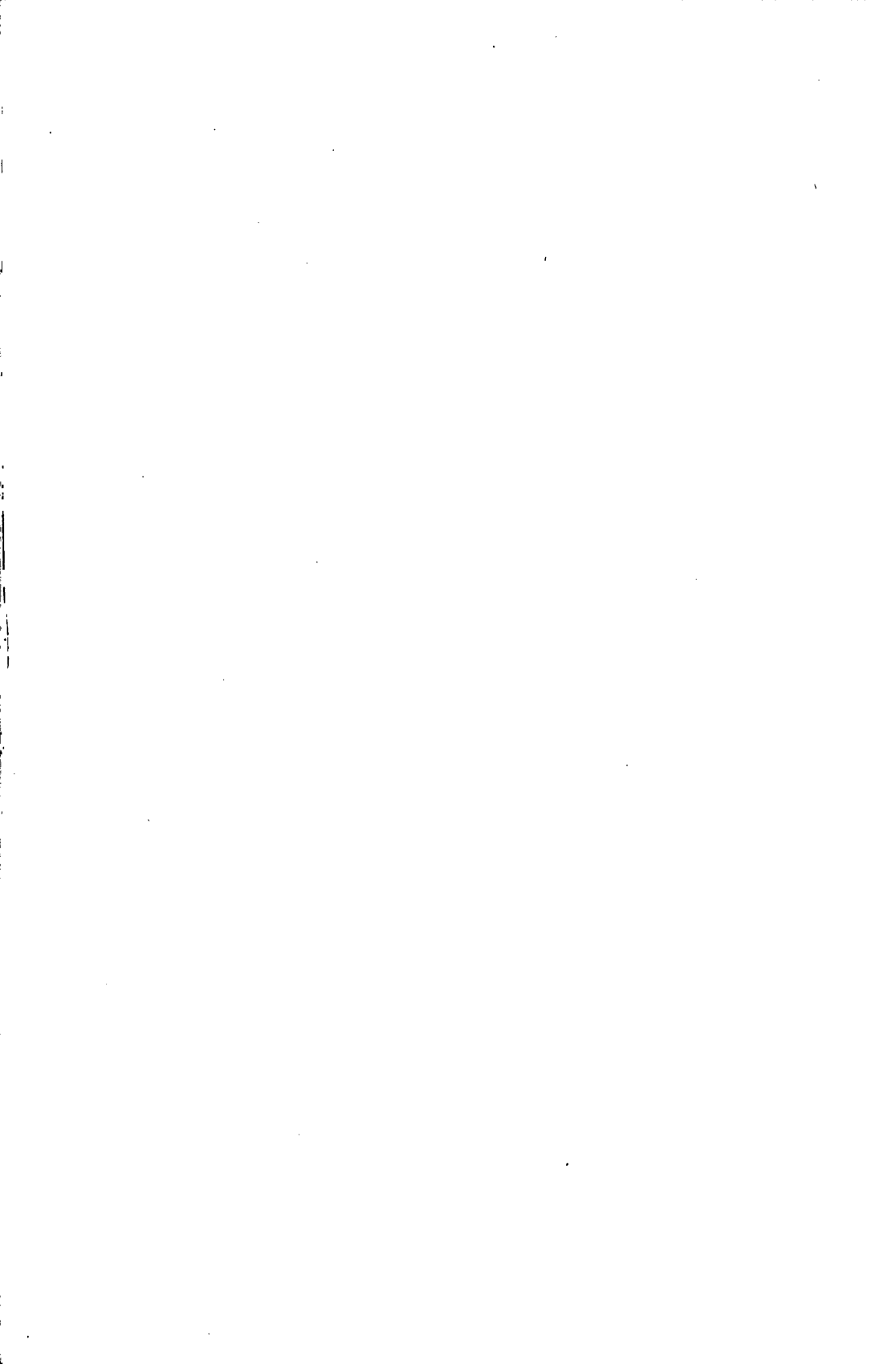
LABORATORY NOTES
ON
INDUSTRIAL WATER ANALYSIS

ELLEN H. RICHARDS

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LABORATORY NOTES

ON

INDUSTRIAL WATER ANALYSIS

A SURVEY COURSE FOR ENGINEERS

BY

ELLEN H. RICHARDS

INSTRUCTOR IN SANITARY CHEMISTRY, MASSACHUSETTS INSTITUTE
OF TECHNOLOGY

FIRST EDITION

FIRST THOUSAND



NEW YORK

JOHN WILEY & SONS

LONDON: CHAPMAN & HALL, LIMITED

1908

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ELLEN H. RICHARDS.

Stanhope Press
F. H. GILSON COMPANY
BOSTON, U.S.A.

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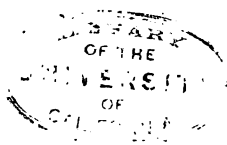
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LABORATORY NOTES ON INDUSTRIAL WATER ANALYSIS.

PART I.

INTRODUCTION.

WATER is needed for many uses, the quality desired varying with the needs of the industry. The quality of water found depends upon the geological formations over which it flows or through which it percolates, and upon the previous use which man has made of it. Because of the growing scarcity of the supply, the increasing use per capita, the congestion of population and the occupation of even the desert and mountain slope, the securing of either safe potable water or water suited to manufacturing purposes becomes more and more difficult, and there is demanded a closer study of the country's resources and of waters suited to the different uses.

Restrictions will undoubtedly be adopted in the near future preventing not only sheer waste and pollution, but assigning various supplies to the most suitable uses. In other words, certain sources of water supply must be saved for the most important needs, and certain other sources must be so treated as to make them usable. Water unsatisfactory for one purpose may be or may be made quite satisfactory for another.

The present generation of engineers may not be confronted with these problems, but the students now in training will cer-

tainly meet them, and should go to them untrammelled by the practice of the past.

Leaving aside potable water, there is a demand for water for steam, for dyeing and textile manufacturing, for brewing, for chemical processes, etc. While each industry has its own peculiar requirements to be determined by the expert chemist, yet the engineer, in deciding upon recommendations, is often required to estimate the value of water for general purposes. It is of great service to him if he is able to reject at once, to classify as good, or to put into the doubtful category the samples he is examining. If this can be done in the field or in the office, so much the better. The one essential point is that the engineer should recognize both his own limitations and those of the method he employs. A trained and experienced chemist may see more meaning in a given reaction than the ordinary observer. A given test may reveal only a part of the truth, or, in unusual circumstances, it may be misleading.

But with all these risks of imperfect work, there are many occasions when a little knowledge is a wholly valuable possession, so that it is worth while for the student to spend thirty hours on a series of experiments which will indicate methods of attack and may save weeks in the future.

Water taken from the deep ground is a sort of residual mother liquor derived from years of time and miles of travel. Surface waters are usually mixed with more or less ground water.

The evaporation of water for steam involves a concentration of whatever the feed water holds in suspension or solution, leaving in the boiler a thick mud or a coating of more or less stony scale. This is a detriment to the efficiency of the boiler as an evaporator and to its strength as well. It follows, therefore,

that a good water for steam production should be fairly free from suspended clay and earth, from silica, and from easily precipitated iron compounds, from calcium and magnesium bicarbonates, which are precipitated on boiling, and from carbonates and sulphates, which are left as a residue on evaporation.

The more friable substances may be removed to a great extent by frequent "blowing off." This involves some waste of power, and involves much inconvenience. The stony substance may be, to a certain extent, converted to a friable condition by chemical reaction, loosened by kerosene, or prevented from adhering by the use of organic substances. The deposition of scale is not the only danger to be guarded against. Certain waters attack the metals composing the boiler shell and tubes, often dissolving so much that essential parts are thereby weakened. Such waters are as uneconomical and dangerous for steam making as are hard waters. Magnesium chloride and nitrates are objectionable for this reason. For manufacturing purposes there are special requirements to be considered under each case.

In this short course only special methods are considered, leaving out the ordinary analytical processes to be found in text-books.

FIRST LABORATORY EXERCISE.

To classify the sample in hand, use the following *preliminary tests*. Note appearance — much or little color, turbidity, sediment, organisms, etc. — as a guide to the final conclusion.

Keep the sample well mixed without violent shaking, to insure uniformity of successive portions taken.

Examine the waters for division into general classes, as follows:

I. Scale-forming,

- (a) Friable deposits: { clay and mud, bicarbonates Ca and Mg; carbonates Ca, Mg, Fe.
- (b) Stony deposits: { chiefly calcium sulphate, or iron carbonate and silica, cementing other substances.

II. Moderate scale-forming, easily controlled,

- (a) Natural waters, not carrying great excess of mineral substances.
- (b) Effluents from alum or iron sulphate filters.

III. Non-scale forming, but corrosive,

- (a) Natural, soft, clear or colored waters, carrying strong acid reaction from CO_2 or "humic" acids. May be high in organic matter.
- (b) Waters carrying magnesium chloride.
- (c) Polluted waters carrying bacterial products, organic or other acids, ammonia, etc.; i.e., rivers acting as sewers.
- (d) Contaminated waters carrying chlorides, nitrates, etc.; i.e., wells and "organic matter free" effluents.

IV. In certain sections of the country another class of natural waters occur carrying sodium salts chiefly, alkaline in character. These cause foaming or priming, and illustrate the care to be taken in the use of sodium carbonate as a remedy. These waters will be indicated by a negative result in titration for permanent hardness. An excess of organic matter also often causes foaming.

Examine the three waters assigned and place them in the proper class, I, II, or III, by comparative qualitative or quantitative tests, for—total hardness, sulphates, carbonates, chlorides, color, carbon dioxide, nitrates, ammonia, and iron, by described methods. For instance, comparison with standards for color and for ammonia; by titration for carbon dioxide, and, if desirable, for organic matter; by evaporation to determine nitrates and iron.

Preliminary "Hardness" Test. 10 cc. of the water is measured into the test bottle *H*, and 40 cc. of distilled water added. From the soap solution burette 1 cc. is run into the bottle *H* (about 6 cm. wide by 12 cm. high to the shoulder), glass stoppered. The contents are well shaken, and if no foam or "lather" remains add successive portions of 1 cc. each, until a partial covering of the surface is noted; then 0.5, and, finally, 0.2 cc. at each addition, until the surface is just covered with a white foam at the end of five minutes, the bottle *H* lying on its side. If not more than 2 cc. of the soap solution have been used, the water contains at most about 97 parts per million of calcium and magnesium salts, and is "soft." Use Table No. II. For a closer determination of soft water make another test, using 50 cc. of the water without dilution, Class III. Use Table No. I.

If 5 cc. of the soap solution have been used, the water contains about 300 parts and is "hard," Class II. If 8 or

10 cc., 500 or 600 parts, and the water is *very hard*, Class I, and no further test is made. If the first foam disappears at the end of three minutes and more is required to secure a permanent (for five minutes) foam, magnesium is indicated. Much magnesium gives also a characteristic curdy scum. If no foam is permanent with 10 cc. of soap solution the water is too hard to be tested by this strength of soap solution. For double strength see Tables III and IV.

In order to gain some knowledge of the samples as an aid in future procedures, from each sample of water take two portions of about 10 cc. each; pour into two test tubes. To the first test tube add a few drops of silver nitrate, AgNO_3 . Note amount and character of the resulting precipitate, then add a few drops of dilute nitric acid, HNO_3 . Note the permanence or disappearance of all or a greater portion of the precipitate. Silver *carbonate* will be dissolved; silver *chloride* will not; thus both carbonates and chlorides are indicated by the same test. If only a slight milkiness remains, the sample must be concentrated (100 cc. or 250 cc. to 25 cc.) before testing for exact amount of chlorine. The water is probably Class III, but is not rigidly classified by this test.

To the second test tube add a few drops of barium chloride, BaCl_2 . Note amount of precipitate, then add a few drops of dilute hydrochloric acid, HCl . If the precipitate perceptibly disappears, carbonates as well as sulphates are present. If a heavy precipitate remains, the turbidimeter may be used with the unconcentrated water, Class I. Otherwise, 200 cc. to 500 cc. are to be put on the water bath to concentrate. If there is no perceptible precipitate on standing, no further test is needed. The water is probably Class II, but is not rigidly classified by this test.

Excess of Carbon Dioxide. Class III or II, rarely I. Titrate 100 cc. of the original water to be tested in a graduated 100 cc. Nessler tube with sodium carbonate to absorb the "free" or excess CO_2 , yielding sodium bicarbonate. If $\frac{n}{50} \text{Na}_2\text{CO}_3$ is used, multiply the cc. required to produce a permanent faintly pink color by 10 to give parts per million of calcium carbonate

~~If $\frac{n}{50} \text{Na}_2\text{CO}_3$ is used, each cc.~~

~~WITH CALCIUM HYDROXIDE~~

water made alkaline with 3 cc. KOH. Holding the tubes close together, run in standard solution from the burette with frequent rotation until the colors match. If more than 25 cc. of the standard is required, discard these solutions and evaporate 1 cc. or even $\frac{1}{2}$ cc. for a new test. On the other hand, if

10 cc., 500 or 600 parts, and the water is *very hard*, Class I, and no further test is made. If the first foam disappears at the end of three minutes and more is required to secure a permanent (for five minutes) foam, magnesium is indicated. Much magnesium gives also a characteristic curdy scum. If no foam is permanent with 10 cc. of soap solution the water is too hard to be tested by this strength of soap solution. For ~~double strength see Tables III and IV.~~

ERRATA.

Page 7, line 8, "g." not "mg."

" 20, " 12, "sulphanilic," not "sulphuric"; "naphthylamine."

" 21, " 20, insert after "tested": "Add 10 cc. sulphuric acid (1:3)."

" 32, " 2 from bottom, "liter," instead of "like."

" " " 1 " " "oxygen." Not "Oxygen."

" " " 1 " " " $\frac{1}{40}$ " begins new sentence.

" 33, " 1, "0," instead of "0°."

" " " 2, "oxygen," instead of "Oxygen."

" 34, " 17, "0.00001 gram Fe."

" " " 18, "*Sulphocyanate*," not "*ide*."

" 35, " 9, insert "a" before "naphthylamine."

" 44, last line, "53.6," instead of "15.6 cc. water."

" 48, line 12, "Eng. Record," instead of "Report."

" " " 20, add after "Society," "Vol. 25, p. 394."

" " " 21, after "Walker," add "Vol. 29, p. 1251."

the unconcentrated water, Class I. Otherwise, 200 cc. to 500 cc. are to be put on the water bath to concentrate. If there is no perceptible precipitate on standing, no further test is needed. The water is probably Class II, but is not rigidly classified by this test.

Excess of Carbon Dioxide. Class III or II, rarely I. Titrate 100 cc. of the original water to be tested in a graduated 100 cc. Nessler tube with sodium carbonate to absorb the "free" or excess CO_2 , yielding sodium bicarbonate. If $\frac{n}{50} \text{Na}_2\text{CO}_3$ is used, multiply the cc. required to produce a permanent faintly pink color by 10 to give parts per million of calcium carbonate equivalent to the excess CO_2 . If $\frac{n}{22} \text{Na}_2\text{CO}_3$ is used, each cc. is equivalent to 0.001 mg. of CO_2 .

Phenolphthalein is used as indicator, and may be added to the sodium carbonate solution when made up.

Determination of Nitrates. With a burette pipette take from each sample (clarified by milk of alumina if turbid or colored) two portions, one of 2 cc. and one of 5 cc., run into 3-inch porcelain dishes; place on the top of the water bath to evaporate for the determination of nitrates. When just dry, cool and add six drops of phenol-di-sulphonic acid. With a short bent glass rod, cause the acid to cover all the residue; add water from the automatic pipette or from a graduate to dilute the acid. Make alkaline and note color, if any. The amount of nitrates present may be estimated by matching this color with the color produced by a known amount of solution, 1 cc. = 0.000001 gram N. Select two tubes of equal diameter, thickness and shade of glass, of 50 or 100 cc. capacity. Rinse the contents of the dish into one of these and make up to the graduation with distilled water. Fill the other tube half full of distilled water made alkaline with 3 cc. KOH. Holding the tubes close together, run in standard solution from the burette with frequent rotation until the colors match. If more than 25 cc. of the standard is required, discard these solutions and evaporate 1 cc. or even $\frac{1}{2}$ cc. for a new test. On the other hand, if

the color in the porcelain dish is very slight it may be matched directly, using a similar dish. The number of cc. of standard used divided by the number of cc. of sample evaporated gives the parts per million.

In the field it may be more convenient to use the Brucine method as follows: to 1 cc. of the water in a 3-inch porcelain dish add 2 cc. concentrated H_2SO_4 ; cool; fold a piece of solid Brucine, about the size of a B.B. shot, in a 7 cm. filter. Place the paper on the side of the dish, washing it with the solution by a rotary motion. The appearance of a bright pink color of greater or less intensity proves the presence of nitrates in greater or less amount. The yellow color which results in a few minutes may be used to compare with standards. This method will detect five parts or more of nitrates per million.

These preliminary tests will show whether the water is *incrustant* or *corrosive*, and will give an approximate idea of the quantities of each important constituent, so that in the quantitative analysis to follow, certain tests may be omitted. For instance, if silver nitrate (solution 25 grams to the liter) causes no perceptible cloudiness, it may be assumed that chlorine exists in less amount than two parts per million. If barium chloride causes no perceptible cloudiness, sulphates are so low as to be neglected for general uses.

If the water is "soft" (less than 100 parts per million), no tests for incrustants need be subsequently applied, but attention may be concentrated on its corrosive qualities.

In Class I if sulphates are predominant, treatment will be required. Treatment with alcohol and the use of the turbidimeter as described on page 10 will probably give good results.

If the chlorides, etc., predominate, other more satisfactory tests are to be applied.

SECOND LABORATORY EXERCISE.

DETERMINATION OF AMOUNT OF SCALE-FORMING MATERIAL IN CLASS I OR CLASS II, AND OF TOTAL SOLIDS.

DETERMINATION OF IRON IN SOME CASES.

DETERMINATION OF SULPHATES BY THE TURBIDIMETER OR OTHERWISE.

Total Solids. Evaporate 100 to 200 cc. of the sample to dryness on a water bath according to observed hardness in a tared platinum dish. Dry in the oven at 100° C. or 110° C. for two hours. Waters high in chlorides absorb moisture very rapidly and dry with difficulty. Cool the dish in a sulphuric acid desiccator and weigh quickly. Use the residue for incrustants or for iron determination.

Incrustants by Non-Solution. Treat the residue three times with 60 per cent alcohol, allowing it to stand 20 minutes each time. Decant carefully after each treatment (it may be necessary to filter if a flocculent precipitate floats, the small ashless filter being held in a twisted wire and ignited over the dish). Dry the dish, cool and weigh as before. This residue gives an approximate per cent, i.e., an *estimate* of the non-soluble material the water will yield on evaporation.

Determination of Iron. If not used for incrustants, the residue from total solids determination is treated with 5 cc. HCl (I-I), warming if solution is not immediate. Wash out into a 100 cc. Nessler tube with distilled water to the 50 cc. mark. Oxidize the possible ferrous compounds to ferric by a few drops of potassium permanganate. The pink color should persist 5 minutes. Make up a blank with 50 cc. distilled water and 1 cc. HCl (I-I). To the sample prepared as above and

to the blank add 15 cc. potassium sulphocyanate, KCys . Place in a good light. To the blank add from a finely graduated 1 cc. pipette, standard iron solution one-tenth cc. at a time, rotating the tube to mix each time, until the red color matches the color of the sample. The standard iron contains 0.00001 gram Fe in 1 cc.

Sulphates by Turbidimeter. The opacity is due to sulphates precipitated as barium sulphate, BaSO_4 , finely divided, and remaining in suspension, unless the sulphates are present in large amount.

Operation. To 100 cc. of water add HCl sufficient to acidify (about 1 cc.) and 1-2 grams BaCl_2 . Shake until dissolved. Pour slowly into the graduated tube of the turbidimeter (*keeping the outside perfectly dry*) until the flame beneath just disappears when looking down through the liquid. *Caution:* The tube must not be placed over the flame when empty. Calculate, after first noting height of liquid in cm. amount of SO_4 , from formula $X = \frac{0.0573}{y + 0.1}$ where X = amount of SO_4 present, y = height of liquid in cm., or from Table No. V. Waters containing 80-300 parts per million read directly; if less, concentrate, if more, dilute before precipitation. The most accurate readings are those obtained in the upper half of the tube.

Determine grams of SO_4 from SO_3 by the ratio of molecular weights. As molecular weight of $\text{SO}_4 = 96$, and of $\text{SO}_3 = 80$, multiply grams of SO_3 obtained by $\frac{96}{80}$, or $\frac{6}{5}$, to obtain grams of SO_4 . The successful use of the turbidimeter requires a little practice and care in securing a steady pointed flame, and careful and frequent mixing of the solution to prevent the heavy precipitate from settling out. Sulphates may also

be determined by the usual gravimetric method, or by titration with potassium chromate (Wehrenfennig, p. 28), as follows: Remove the bicarbonates from 200 cc. of the water to be tested by boiling 15 minutes, taking care to keep the volume by replacing the loss with distilled water, to prevent the precipitation of calcium sulphate by concentration. Decant, or filter 100 cc. into a graduated 150 cc. flask; add 10 to 20 cc.

$\frac{n}{10}$ barium chloride, according to amount of sulphate present.

Heat 5 minutes and add from a burette $\frac{n}{10}$ potassium chromate until the supernatant liquid shows a faint but distinct yellow. Cool, fill to the mark with distilled water, filter 100 cc. into a Nessler tube. Make a blank with distilled water in a companion tube, running in from the burette sufficient potassium chromate to match the color. The difference between the number of cc. used with the sample and with the blank, multiplied by $\frac{3}{2}$, subtracted from the number of cc. of barium chloride used, multiplied by 40, gives parts per million.

THIRD LABORATORY EXERCISE.

Alkalinity (including "temporary hardness") comprises the carbonates and hydrates which will react with added sulphuric acid. It is frequently necessary to determine this before deciding upon treatment.

Magnesium as hydrate, set free by calcium hydrate, may be determined in the same solution.

Permanent Hardness. Calcium and magnesium sulphates which will react with added sodium or potassium carbonates or hydrates, but which is not removed without such reaction.

Alkalinity by Titration. Measure 100 cc. of the sample into a 200 cc. graduated flask and titrate with $\frac{n}{50}$ H_2SO_4 , using methyl orange as an indicator. The number of cc. of acid used multiplied by 10 gives parts per million calcium carbonate corresponding to the alkalinity. This value multiplied by 3.42 gives the weight of aluminum sulphate $\text{Al}_2(\text{SO}_4)_3$ which the water will decompose, and by 4.74 gives the weight of alum, $\text{KAl}(\text{SO}_4)_2 \cdot 12 \text{H}_2\text{O}$. When lacmoid is used as an indicator, measure 100 cc. into a 6-inch porcelain evaporating dish; add 0.5 cc. of lacmoid solution (2 grams in one liter of 50 per cent alcohol). Heat until nearly boiling, run in the $\frac{n}{50}$ sulphuric acid, as before, until a reddish-purple takes the place of the blue color. Heat again, and if the blue returns drop the acid cautiously into the middle of the dish, noting any change in color as the drop spreads. Read the burette for the total number of cc. used.

When heat is not desirable or available, and methyl orange is objected to, 100 cc. may be measured into a 250 cc. white glass-stoppered bottle, 2.5 cc. erythrosine solution (0.1 gm. of the sodium salt in one liter of distilled water), added together with 5 cc. of chloroform (neutral to erythrosine), and the $\frac{n}{50}$ acid run in a few drops at a time. The bottle must be shaken vigorously. The rose color should slowly disappear until a white paper held back of the bottle fails to reveal a trace of pink in the liquid above the chloroform.

For *magnesium*, use the already titrated sample by boiling for 15 minutes in the flask, then adding from a burette (closed circuit) 25 cc. or for waters high in magnesium 50 cc. saturated "lime water," calcium hydrate, and allow to stand on the water bath or hot plate 15 minutes longer. The 200 cc. flask should have an additional graduation at 205 cc. to allow for the expansion of the liquid from 17°–100° C. At the end of the 15 minutes, fill to the 205 cc. mark with boiling distilled water, mix and filter quickly into a graduated cylinder, and reject the first 30 or 40 cc. (used to wash the filter paper and to heat the cylinder). Titrate the next 100 cc. with $\frac{n}{50}$ H_2SO_4 , using methyl orange as indicator. Since with the most rapid and careful manipulation a certain carbonation of the "lime water" may occur, a blank is put through all the operations including filtration with the same number of cc. of the calcium hydroxide (if several samples are done at the same time, one blank will serve). The difference between the acid used for the blank and for the sample is the number of cc. which would have been used up by the lime water which has entered into combination with the acid radical and driven out the magnesium as hydrate, a flocculent precipitate.

Because only 100 of the original 200 cc. is used, multiply by 20 the cc. of acid used to obtain the parts per million of calcium carbonate equivalent to the magnesium in the sample. The ratio $\text{CaCO}_3 : \text{Mg} :: 100 : 24.18$ will give parts per million Mg.

The whole operation should not occupy more than forty-five minutes, including the two fifteen-minute periods of heating.

To Determine Magnesium by Soap Titration. To 100 cc. of water found to contain magnesium add 0.1 gram powdered ammonium oxalate. Shake until dissolved and filter. Test 50 cc. with the soap solution. The calcium should have been removed as oxalate.

For Permanent Hardness (incrustants par excellence). Boil in a porcelain dish 250 cc. of the water. Add 25 cc. of $\frac{n}{10}$ "soda reagent" (made of equal parts of sodium hydroxide and sodium carbonate. This mixture is especially for magnesium waters), and boil for 10 minutes. For waters which show a hardness by the soap test of over 400 parts per million use $\frac{n}{2}$ soda reagent. Filter, and while hot make up with boiling distilled water to 250 cc.; mix and titrate 100 cc. with $\frac{n}{50}$ H_2SO_4 , using methyl orange as indicator.

Make a blank, using boiling distilled water instead of the sample. The difference gives the cc. "soda reagent" used up by the permanent hardness. This number of cc. multiplied by ten gives the parts per million of calcium carbonate corresponding to permanent hardness, $\text{CaCO}_3 : \text{CaSO}_4 :: 100 : 138$. If the water shows excess of "soda reagent" over the blank, it contains sodium or potassium carbonate, and the number of cc. multiplied by 10 gives parts per million of calcium carbonate equivalent to the sodium carbonate present.

Those students who have samples very "hard" with carbonates or sulphates may use the following method for incrustants. It is not so satisfactory for chloride or nitrate waters.

I. *Determination of Scaling Material.* By means of a 250 cc. graduated flask, measure 250 cc. of the water to be examined into a 6-inch porcelain evaporating dish. Then add, from a burette or 10 cc. pipette, exactly 10 cc. of $\frac{n}{10}$ sodium carbonate solution. Place on the hot plate, or if short of time place over the flame of the burner and keep gently boiling until the volume is reduced to about 25 cc. Be careful not to boil too hard, or some of the water will be lost by spurting. While the water is being evaporated fold two 11 cm. ashless filter papers and put into the drying oven; after one hour remove them, cool in the desiccator and counterpoise them on the balances, cutting from the heavier until they are just equal in weight. Also dry and weigh a No. 2 or a No. 3 beaker at the same time.

Put the filter papers into a funnel, one inside the other, and stand the weighed beaker under to catch the filtrate. The end of the funnel should touch the side of the beaker so that the liquid will not spatter out on dropping. Next, filter the material in the evaporating dish through the papers, washing down all loose precipitate with a fine jet of water from the wash bottle, using as little water as possible. To clean the dish add 5 cc. of $\frac{n}{10}$ hydrochloric acid from a clean burette and rub over the surface of the dish; add 10 cc. of $\frac{n}{10}$ sodium carbonate from the pipette and boil for one minute. Turn the dish so that the solution touches all parts of its surface, exactly neutralizing the hydrochloric acid. Filter through the same papers, rubbing the dish with a rubber-tipped stirring rod until

every particle of precipitate is loosened. Wash into the funnel with hot water again, using very little water.

Wash the filter and its contents until it is free from soluble matter, requiring about six applications of water. The filtrate and washings in the weighed beaker now contain the soluble alkalies, and the precipitate comprises all the scale-forming material. Dry the latter in the papers at 110° C. in the oven, cool in the desiccator and weigh, putting the empty (tared filter) paper on the right-hand pan.

(This method is used by Professor Parr of the University of Illinois.)

FOURTH LABORATORY EXERCISE.

Waters belonging to Class III especially may be tested for their action on metals. Those that show the presence of ammonia in the preliminary test may be distilled for quantitative determination.

Suspected waters may be tested for nitrites and chlorine, concentrating if necessary, to compare with a known normal.

The amount of oxygen absorbed from potassium permanganate (oxygen consumed) which gives a certain indication of the amount of organic matter is often a useful determination. In some cases "oxygen dissolved," or that which the fishes breathe, yields valuable information.

Action on Metals. For class illustration it will be sufficient to use freshly polished specimens of the common metals, lead, copper, brass, iron and steel of different grades. These placed singly in sufficient water to cover them an inch deep in tubes or in filled stoppered bottles (the size and number being somewhat regulated by the amount of water at hand), may be observed hour by hour and at the end of 12 and 24 hours. The water after filtration may be tested by appropriate methods.

The following tests modified from those given in Thresh's book will serve most conveniently.

Lead. Decant 5 to 50 cc. of the water in which the metal has been standing into a test tube; add two drops to 1 cc. of acetic acid I:I; mix, and with a capillary tube or a glass rod add "a droplet" of calcium sulphide (see Reagents, p. 35). Note carefully against a white background, holding tube with opera-

tor's back to the light. The white sulphur separates out on contact with the acid solution and turns dark, or if much lead be present, black. If the first addition shows nothing, make a second or third under the same careful observation.

To make this and the following tests quantitative, measure the original solution accurately into wide Nessler tubes and determine by means of standard solutions.

This test will detect 1 part lead in 10,000,000 of water. By passing hydrogen sulphide gas through the solution a considerably greater delicacy has been obtained, or the water may be concentrated, after the addition of nitric acid, subsequent neutralization with KOH, and acidification with acetic acid (which does not decompose H_2S as does nitric acid), and the test made as before.

Lead sulphide is black even in small quantity, copper sulphide is copper brown, and therefore if either are present alone they may be detected, but if both are present in the same sample the one color obscures the other. The mixed sulphide may be oxidized by a drop of strong nitric acid, lead precipitated by a drop of sulphuric acid and ammonia added to alkalinity. If a trace of copper be present a characteristic blue color is produced.

Copper. Decant as before, add 1 drop to 1 cc. (according to the amount of water used) of H_2SO_4 (1:4), mix, add 1 drop to 1 cc. of a freshly made potassium ferrocyanide solution.

A copper-brown color indicates the presence of copper which may be quantitatively determined by means of standards. Lead does not interfere, but iron, if present, must be removed by precipitation with ammonium hydrate and the filtrate acidified and tested.

Zinc. Decant and treat precisely as in the test for copper. A white turbidity or milkiness indicates the presence of zinc,

becoming opaque white if the zinc is abundant. A dilution which shows turbidity only may be matched with standard solutions.

Considerable quantities of copper or iron interfere and must be removed.

Iron. Decant and acidify as before, but oxidize the ferrous iron to ferric by adding potassium permanganate (reagent) until a distinct pink color remains for two minutes, then add the ferrocyanide.

A blue color indicates the presence of iron, and its depth is proportional to the quantity of iron, which may be determined by means of standards.

If zinc is present, the turbidity may interfere with accuracy.

For the Determination of Free and Albuminoid Ammonia. Free the flask and condensing apparatus by distilling water until there is no test with Nessler reagent. Empty the flask, and without rinsing measure into it 100 cc. of badly polluted waters, or 500 cc. of ordinary quality. If a smaller quantity is used, make up to 500 cc. with ammonia-free water. If the water has an acid reaction, neutralize by sodium carbonate before distilling. Collect 3 portions of 50 cc. each; allow the flask to cool ten minutes, and add 40 cc. alkaline permanganate through a funnel to prevent the least drop from touching the neck of the flask whence it would contaminate the cork or rubber stopper. Watch until boiling has fairly set in to prevent foaming or bumping, lest the stopper be touched by spatters. Collect 3 or 4 portions of 50 cc. each as before. When all are ready prepare standards or use permanent ones. Nesslerize with 1 cc. of the reagent and compare with standards. The first three tubes give the ammonia readily disengaged, "free ammonia," and the last set that which has to be broken out of

combination, so to speak, from as yet undecomposed organic matter, "albuminoid ammonia."

The sum of the number of cc. of standard used to match the different tubes, multiplied by 0.02, gives parts per million free or albuminoid ammonia as the case may be.

Without distillation the student may sometimes mistake the yellow color given by the potassium hydrate of the Nessler with iron for the mercur-ammonium compound indicating ammonia. *Nitrites* with ammonia indicate active pollution and therefore a probable corrosive action.

Procedure. To 100 cc. of sample in a Nessler tube add 10 cc. each of sulphuric acid and naphthalamine acetate. Allow to stand ten minutes and compare color with standard papers.

Quantitative Determination of Chlorine (Air, Water and Food, p. 111). Measure 25 cc. of the water to be tested into a 6-inch evaporating dish; add 6 drops of neutral potassium chromate and titrate with AgNO_3 , 1 cc. = 0.0005 Cl, approximately. The first rosy flush must be used as the end point. 250 cc. of the water must be concentrated to 25 cc. if it contains less than 20 parts Cl per million.

Oxygen Consumed (Absorbed).

All waters when strongly acidulated with sulphuric acid and digested with a little permanganate of potash absorb from this salt more or less oxygen, the amount of which can be determined if the amount of available oxygen in the permanganate added is known and the amount left after the action of the water is determined. The difference gives the oxygen absorbed by the substances dissolved in the water. Some very pure waters absorb very little indeed, less than 0.1 mg. per liter, while others containing organic matter in solution absorb many times this

amount. Although, strictly speaking, an index neither to the quantity nor quality of the organic matter, yet, as the amount absorbed varies in different waters, being usually very small in pure waters and comparatively large in impure waters, the determination is not without value. Certain inorganic substances occasionally found in waters also reduce permanganates, such as nitrites, ferrous salts, and sulphides. These act on the permanganate with rapidity, while the organic matter acts very slowly. When any of these substances are present, two determinations are generally made, one to ascertain the amount of oxygen absorbed by the inorganic matter, and the other to estimate the total absorbed oxygen, and the difference is taken as being the amount consumed by the organic matter. The total oxygen consumed varies greatly in the same water, the chief factors being time and temperature; but the degree of acidity and the intensity of the light are not unimportant. For results to be comparable, therefore, they must have been obtained by identical processes.

Oxygen Consumed: Quick Method. Measure into a 150 cc. flask 25, 50 or 100 cc. of the water to be tested. ^{add 10 cc. $H_2SO_4(1-2)$} Add from a burette 8 to 15 cc. of the standardized permanganate. Bring to a boil, and boil two minutes. Cool one minute; add from a burette 10 cc. (or more if this amount does not decolorize the solution) of ammonium oxalate. Titrate with the permanganate to a faint pink. The difference between the total amount used and that given by a blank determination gives the oxygen consumed.

Oxygen Consumed: Dr. Thresh's Method. Apparatus and reagents required: Standard solution of potassium perman-

ganate, 1 cc. = .1 mg. available oxygen. Solution of sodium thiosulphate, 1 gram to the liter. Solutions of potassium iodide and of starch. Solution of sulphuric acid, 25 per cent. Stoppered bottles or flasks holding about 400 cc. Burettes, pipettes, etc. Two hundred and fifty cc. of water to be examined heated to 98° F. are measured into one of the bottles or flasks, which should have been previously cleaned with acid, etc. To this are added 10 cc. of the solution of potassium permanganate and 10 cc. of the sulphuric acid, and the stopper being inserted the bottle is placed in an incubator kept at about 98° F. Let it remain there for three hours, examining it from time to time to see that a decided pink color remains. If the color tends to disappear, add a second 10 cc. of permanganate solution, as this should always be present in marked excess. While this is incubating, place 250 cc. of the recently distilled water in a second flask, add 10 cc. of the acid, 10 cc. of the permanganate, and 1 cc. of 5 per cent potassium iodide solution, using starch as an indicator. The amount of the solution used corresponds to 1 mg. of available oxygen, or to 10 cc. of the permanganate solution. The thiosulphate solution not keeping well, this standardization should be repeated with every fresh batch of waters or every few days. On no account should the thiosulphate solution be made with a water containing nitrates, for, if so, nitrites will be formed and vitiate the experiment.

The water, after the lapse of three hours, is removed from the incubator, and quickly reduced to the room temperature by immersing the bottle in cold water. The iodide is then added, and the excess of permanganate estimated. In this determination it is most important to cool the water, as the amount of thiosulphate required to destroy the blue color of the iodide of starch is markedly affected by the temperature. This is

another of the causes, not generally recognized, of the differences in the amount of oxygen absorbed found by different analysts when examining the same water. If it is desired to estimate the oxygen absorbed by the inorganic matter, the water may be warmed to 98° F., and the un-reduced permanganate estimated. In examining potable waters this determination is rarely required.

Oxygen Dissolved.

Collect a sample of the water in a calibrated bottle of about 250 or 300 cc., taking care that no air is inclosed. This can be most readily done by allowing water to flow into the bottom of the bottle by means of a piece of rubber tubing attached to glass running through the neck. Then allow the water to overflow for some minutes, at the same time noting the temperature of the water.

Procedure for Oxygen Dissolved. Remove the stopper from the bottle and add approximately 2 cc. of the manganous sulphate solution and 2 cc. of the sodium hydrate-potassium iodide solution, delivering both of these solutions beneath the surface of the liquid by means of a pipette. Replace the stopper and mix the contents of the bottle by shaking. Allow the precipitate to settle. Remove the stopper, add about 2 c.c. of sulphuric acid and mix thoroughly. Up to this point the procedure may be carried on in the field, but after the sulphuric acid has been added and the stopper replaced there is no further change, and the rest of the operation may be conducted at leisure. For accurate work there are a number of corrections necessary to take into account, but in actual practice it is seldom necessary to take note of them, as they are

ordinarily much less than the errors of sampling. Rinse the contents of the bottle into a flask, titrate with $\frac{N}{40}$ solution of sodium thiosulphate, using a few cc. of the starch solution towards the end of the titration. Do not add the starch until the color has become a faint yellow; titrate until the blue color disappears.

Calculation of Results (taken from Standard Methods). The standard method of expressing results shall be by parts per million of oxygen by weight.

"It is sometimes convenient to know the number of cc. of the gas per liter of 0° C. temperature and 760 mm. pressure, and also to know the percentage which the amount of gas present is, of the maximum amount capable of being dissolved by distilled water at the same temperature and pressure. All three methods of calculation are therefore here given:

$$\text{Oxygen in parts per million} = \frac{0.0002 N \times 1,000,000}{V} = \frac{200N}{V}$$

$$\text{Oxygen in cc. per liter} = \frac{0.1395 N \times 1000}{V} = \frac{139.5 N}{V}$$

$$\text{Oxygen in per cent of saturation} = \frac{200 N \times 100}{V \times O} = \frac{20,000 N}{VO}$$

Where N = number of cc. of $\frac{N}{40}$ thiosulphate solution,

V = capacity of the bottle in cc. less the volume of the manganous sulphate and potassium iodide solution added (i.e., less 4 cc.).

O = the amount of oxygen in parts per million in water saturated at the same temperature and pressure."
(See Table A.)

TABLE A.

QUANTITIES OF DISSOLVED OXYGEN IN PARTS PER MILLION BY WEIGHT
IN WATER SATURATED WITH AIR AT THE TEMPERATURE GIVEN.

Temp. C.	Oxygen.	Temp. C.	Oxygen.	Temp. C.	Oxygen.	Temp. C.	Oxygen.
0	14.70	8	11.86	16	9.94	24	8.51
1	14.28	9	11.58	17	9.75	25	8.35
2	13.88	10	11.31	18	9.56	26	8.19
3	13.50			19	9.37		
4	13.14	11	11.05	20	9.19	27	8.03
5	12.80	12	10.80	21	9.01	28	7.88
		13	10.57			29	7.74
6	12.47	14	10.35	22	8.84	30	7.60
7	12.16	15	10.14	23	8.67		

FIFTH LABORATORY EXERCISE.

REMEDIES DEVISED AND TESTED FOR THE DEFECTS FOUND.

Waters most seriously objectionable for use in boilers are apt to contain the following substances:

Substances which form scale:

Calcium sulphate.

Magnesium and calcium carbonates.

Soluble salts: oxides of iron, silica, and aluminum.

Clay and sand.

Organic matter as cementing material.

Substances which corrode boilers:

Acids, both mineral and organic.

Magnesium chloride.

Certain organic matter.

Substances which cause priming:

Sodium and magnesium carbonates.

Certain organic matter.

Alkalies.

Remedies for the defects found in waters are both general and specific. "Hard" waters are softened by removal of the calcium salts by precipitation and filtration. Any inexpensive substance that will accomplish this without rendering the water too corrosive is a "remedy." Treat 100 cc. of the sample with a known quantity of the substance chosen, "soda ash," sodium carbonate, tri-sodium phosphate, sodium fluoride, etc. After the reaction is complete (it is usually hastened by heat,

when the original volume must be made up by distilled water), filter 50 cc. and test for hardness, etc.

After treatment, determine if the action of the water on metals has been increased or diminished.

Class II may require very careful study to determine whether frequent blowing off and greater care are not cheaper than reagents. If only two or three fillings of the boilers can be used before wasting it ought to be cheaper to set up a water-softening plant.

Class III frequently needs treatment from another reason,—accumulation of sludge, foaming or priming, corrosion or need of removal of color and turbidity for manufacturing. The latter is usually accomplished by a substance like alum, which, decomposed in the water by some alkaline substance, forms a gelatinous drag net and carries down, if allowed to settle out, the objectionable clay and color as well as germs. The waters which show sufficient alkalinity to decompose alum cake, aluminum sulphate, may be tested for the lowest limit of alum which will do the work, because each grain per gallon of *anything* added to water makes an expense which soon becomes prohibitive. For this reason laboratory tests for remedies are at best only indicative of the general direction in which to work, and they serve as a preventive of serious mistakes and as time savers in suggestion of means.

Patent "boiler compounds" are not to be recommended. Analyses of the scale formed in any given case will often give light. See scheme at end of book.

Pitting is probably largely due to inequalities in the composition of the metal. Studies on these lines are in progress.

A preliminary test frequently useful is the behavior of the sample on simple heating and on heating with "correctives" such as "soda ash" or sodium phosphate. Heat to boiling

on the iron plate about 50 cc. in a small Erlenmeyer flask. Note if precipitation occurs indicating escape of excess of CO_2 , or if foaming occurs as concentration takes place. Add a little sodium carbonate in powder (soda ash). Note if sulphates precipitate $\text{CaSO}_4 + \text{Na}_2\text{CO}_3 = \text{CaCO}_3 + \text{Na}_2\text{SO}_4$. To the boiling sample add tri-sodium phosphate. Note precipitate. $\text{CaCO}_3 + \text{H}_2\text{O} + \text{CO}_2 = \text{Ca}(\text{HCO}_3)_2$. Primary or acid calcium carbonate is formed when normal carbonate is dissolved in water containing carbon dioxide. It cannot be isolated, and it decomposes at boiling temperature. CaCO_3 is precipitated and CO_2 escapes.

A statement was made in the *Railroad Gazette*, March 23 and 30, 1900, that outside of New England and some parts of the Middle South there are very few places where pure soft water can be obtained for locomotive boilers: "In the former localities boilers are found to be in good condition after twenty-five to thirty years' continuous service, while in bad water regions most of the boiler parts have to be renewed every five years or oftener. From statistics gathered the estimate is made that each locomotive using bad water is an expense of \$750 annually. The only way to prevent incrustation and corrosion is to purify the water before it enters the boiler: any process or method by which water free from these impurities can be delivered to the locomotive at less expense than fifty cents per one thousand gallons will be an advantageous arrangement."

This illustrates only one phase of the modern problem of securing the right quality of water for a given use.

PART II.

STANDARD SOLUTIONS.

"*Standard*" *Solutions* are made of any convenient value, provided it is known. Thus it is convenient to have a salt solution of one milligram, 0.001 gram per cc. instead of a normal or even a tenth normal $\frac{n}{10}$ NaCl. If the value is known it is standard whether normal or not. C. P. chemicals are used.

The molecular weight of NaCl is $23 + 35.5$, and *Normal* solution would contain 58.5 grams per liter, or 0.058 per cc., and of chlorine 0.0355, which is 35 times too much for use in this case, therefore $\frac{58}{35.5} = 1.648$ grams per liter are weighed out to give a value to 1 cc. of 0.001 gram Cl, a *Standard* solution.

The molecular weight of sulphuric acid, H_2SO_4 , is $2 + 32 + 64 = 98$, but a normal solution is one containing one hydrogen equivalent, therefore a *normal* sulphuric acid solution contains 49 grams H_2SO_4 in the liter. $\frac{n}{10} \cdot \frac{n}{50}$ or any desired strength is made by the suitable dilution. But for the determination of excess of carbon dioxide a standard solution of $\frac{n}{22}$ is used to give a direct reading of 1 cc. = 0.001 CO_2 . Sulphuric acid, H_2SO_4 , 98 grams, unites with Na_2CO_3 , 106 grams, or displaces $12 + 32 = 44 CO_2$. Since it is twice the normal, 22 grams of CO_2 are to be used. The calculation is 1 cc. = $0.022 \cdot \frac{n}{22} =$
1 cc. $\frac{n}{22} H_2SO_4 = 0.001$ gram CO_2 .

Standard solutions are used not only with indicators to show when the desired reaction is completed, but also to produce colors which may be used in comparison with those in solutions of which the value is to be determined.

There are many substances the quantity of which may be estimated by the depth of color they give to the water in which they are dissolved. Picric acid and potassium chromate, for instance, dissolve with a characteristic color which is deeper the more of the substance present.

The quantitative determination is always made by the use of standard solutions explained above in which *known* quantities under *comparable* conditions are matched in color with the unknown. This method of estimating quantities of dissolved substances is of the greatest use in water analysis where the amounts are usually so small as to necessitate the evaporation of large volumes to secure a workable concentration. The earlier practice required weeks of time and gallons of water to perform an analysis which may now be completed with equal or greater accuracy in a few hours, using a few centimeters of water.

Substances liable to change during prolonged heating may now be determined in a few moments. Only spectroscopic methods exceed in delicacy some of the well-known colorimetric tests; for instance, by Nessler's reagent the ten-thousandth of a per cent of ammonia may be detected; a thousandth of a per cent of nitrite may be determined in ten minutes. In both these cases, the substances are so liable to change that the concentration of large amounts of solution would be impossible.

The apparatus required for colorimetric tests is usually very simple, easily carried and cleaned. Some tests, however, require

expensive instruments of precision called tintometers or colorimeters. These may save in time what they cost in money.

There are several limiting conditions to the use of colorimetric methods which must be clearly understood at the outset.

The depth of color given by a definite quantity of a substance is sometimes affected by temperature, as in the case of the ammonia determination, or by the presence of other substances, as in the nitrite test.

One of the most remarkable changes occurs with the dissociation of many substances on reaching a certain degree of dilution. The ions of many substances have a different color from the compound. Thus copper sulphate changes from blue to green on dilution; the mercur-ammonium from red to yellow. The dilution used is often, in fact usually, such that a mixture of colors results. From this it is seen at once that the standard used must be quite comparable in all respects.

The manner in which the light strikes the solution, the kind and thickness of glass used to contain it, the color of surrounding objects, all have an influence on the tint of color seen, and therefore all these must be the same for the standard and for the unknown solution. Moreover, since color is a subjective phenomenon due to the action of light on the eye itself, no two individuals see precisely the same color effect in the same substance, or can describe accurately what they see.

This fact makes the use of so-called *permanent* standards of less value for use by different persons than standards in gravimetric tests.

In cases where no great degree of accuracy is demanded, as in the determination of nitrites in water (time and the surrounding atmosphere affect the result greatly), standard papers may be used advantageously. Colored glass is extensively employed

for comparison, and only its expense prevents a wider use. Some metallic compounds keep the color and bear dilution sufficiently well to be used, but the student should know how to prepare standards for himself from comparable solutions, and should bear in mind the limits as above noted, and should watch for others which may occur, thus finding his personal equation in color work. There is a decided choice as to which depth of color may be compared with the greatest accuracy; for instance, that given by 1 to 3 cc. of standard ammonium chloride solution is more easily differentiated than a color given by 8 to 12 cc., and the color given by 5 cc. of standard nitrate in 10 cc. volume is more easily matched than a deeper color. As a rule, the lighter shades are preferred. The dilution is made before the reaction is brought about, in most cases, by accurately graduated measuring vessels with complete mixing of the liquids.

A few substances will dilute proportionately after the color is produced, as picric acid in the nitrate test, but the color produced by Nessler reagent in the ammonia test will not so dilute. Most of the colors change on standing, and therefore the test is carried out at once.

Many solutions do not retain their strength in the light, or in presence of organic matter, or because of molecular decomposition, as potassium permanganate, silver nitrate, sodium thiosulphate; such solutions need frequent standardization; for example, sodium thiosulphate in the determination of dissolved oxygen is standardized by potassium bichromate solution: $K_2Cr_2O_7 + 14 HCl + 6 KI = 8 KCl + 2 CrCl_3 + 7 H_2O + 6 I$. The molecular weight is therefore six times the "normal" based on hydrogen = 1.

49.07 grams is the weight for a like "normal" bichromate and will yield $\frac{30 \text{ or } 48}{6}$ 8.000 grams. ^{liter} Oxygen, $\frac{1}{40}$ normal will yield

0.200 gram O_2 and 1 cc. (1000 cc. to the liter) will yield 0.0002 gram or 0.1395 cc. O_2 at $0^\circ C.$ and 760 mm.

Oxygen 16 : Iodine 127 :: 0.0002 : x 0.0016.

$\frac{1}{40}$ normal thiosulphate (6.2 grams to the liter) should also give 0.0002 gram oxygen per cc. if of standard strength. To test it: Measure out 10 cc. of the bichromate from a burette pipette into a flask. Add 3 cc. KI, 3 cc. strong HCl, 100 of water, and shake two or three minutes. Titrate the liberated iodine with the thiosulphate to be tested. Note how much weaker or stronger it is and use the correction in the calculation.

The keeping qualities of the thiosulphate solution are improved by adding to each liter 5 cc. of chloroform and 1.5 grams of ammonium carbonate before making up to the prescribed volume.

Oxygen Dissolved.

Standard thiosulphate 1 cc. = 0.0002 gram oxygen. Standard bichromate, see above.

Standard Ammonium Oxalate Solution. Dissolve 0.888 gram of the substance in one liter of distilled water. 1 cc. is equivalent to 0.0001 gram of oxygen.

Reagents:

- (1) Manganous sulphate solution, 48 grams $MnSO_4$ in 100 cc. distilled water.
- (2) Sol. of NaOH and KI. Dissolve 360 grams NaOH and 100 grams KI in 1 liter of distilled water.

Hardness.

Standard Soap Solution. Dissolve 100 grams of the best white castile soap in a liter of 80 per cent alcohol. Dissolve 75–100 cc. of this in about a liter of 70 per cent alcohol, or until 14.25 cc.

of it give the required lather with 50 cc. of standard calcium chloride solution.

Standard CaCl_2 Solution. Dissolve 0.2 gram pure Iceland spar or CaCO_3 in dilute HCl . Evaporate to dryness several times to remove excess of acid. Dissolve in 1 liter of water.

Nitrates.

1. *Standard Nitrate Solution.* Dissolve 0.720 gram pure recrystallized KNO_3 in 1 liter of water. Evaporate 10 cc. of this cautiously on water bath. Moisten quickly and thoroughly with 2 cc. of phenol-disulphonic acid and dilute to 1 liter. 1 cc. = 0.000001 gram N.

Reagent.

2. *Phenol-disulphonic Acid.* Heat together 3 grams synthetic phenol with 37 grams pure concentrated H_2SO_4 in a boiling water bath for 6 hours.

3. *Brucine.* H_2SO_4 concentrated.

Iron.

Standard Iron Solution. Dissolve 0.86 gram of ferric ammonium alum in 500 cc. of water, add 5 cc. HNO_3 (Sp. Gr. 1.20) and dilute to 1 liter. 1 cc. = ~~0.00001~~^{0.00001} gram Fe.

Reagents.

Potassium Sulphocyanate^{ate} 5 grams per liter.

Potassium Permanganate. 5 grams per liter.

Magnesium.

Saturated Lime Water. Shake 1 part of freshly slaked lime with 20 parts of distilled water for 20 minutes and let solution stand over night. Keep in bottle with closed circuit.

Nitrites.

1. *Standard Nitrite Solution.* Dissolve 1.1 grams specially prepared silver nitrite in water. Precipitate silver with NaCl solution for Cl determination, and make up to 1 liter. 100 cc. of this are diluted to 1 liter, and 10 cc. of this last solution diluted to 1 liter gives the standard solution. 1 cc. = 0.0000001 gram N.

2. *Sulphanilic Acid.* Dissolve 3.3 grams sulphanilic acid in 750 cc. of water by aid of heat; add 250 cc. glacial acetic acid.

3. *Naphthylamine Acetate.* Boil 0.5 gram of naphthylamine in 100 cc. of water for 5 minutes. Filter through a plug of washed absorbent cotton. Add 250 cc. glacial acetic acid and dilute to 1 liter.

Chlorine.

1. *Standard Salt Solution.* Dissolve 16.48 grams of fused NaCl in 1 liter of distilled water. Dilute 100 cc. of this to 1 liter for standard. 1 cc. = 0.001 gram Cl.

2. *Standard Silver Nitrate.* Dissolve about 2.42 grams AgNO_3 (dry crystals) in 1 liter of distilled water. 1 cc. = 0.0005 gram Cl approximately. Standardize against the NaCl solution.

Oxygen Consumed.

1. *Dilute Sulphuric Acid.* One part sulphuric acid to three parts of distilled water. This should be freed from oxidizable matters by adding potassium permanganate until a faint pink color persists after standing several hours.

2. *Standard Potassium Permanganate Solution.* Dissolve 0.4 gram of the crystalline compound in 1 liter of distilled water. Standardize against an ammonium oxalate solution. One cc. is equivalent to 0.0001 gram of available oxygen.

Reagent for Metals.

Solution of Ca sulphide.

Sulphur in fine powder.....	20 grams.
Slaked lime.....	20 grams.
Distilled water.....	500 cc.

Boil until about 400 cc. remain.

The solution should have a rich orange-red color; when this fades the solution has become useless.

Solution of Potassium Ferro-Cyanide. Dissolve a clear yellow crystal as large as a large pea in 25 cc. of distilled water.

This solution does not keep, and must be made up fresh on the day it is to be used.

COMPUTATION OF HYPOTHETICAL COMBINATIONS FROM ANALYSES.

The business man always asks for the compound known to him in other relations to be reported from a water analysis. This is not often possible with any degree of exactness in the limitation of our present knowledge. In the case of medicinal water, which was the earliest form of analysis, the combination of the mineral substances was doubtless of particular consequence.

SAMPLE REPORT.

RESERVOIR WATER FROM CYPRESS, ILLINOIS, AUGUST 1 TO 30, 1906.

Ions.	Parts per Million.	Hypothetical Combinations.		
		Ions.	Parts per Million.	Grains per Gallon.
Turbidity.....	146.	Sod. nitrate..(NaNO_3)..	1.1	.06
Susp. solids.....	58.1	Sod. chloride (NaCl)..	11.2	.65
Diss. solids.....	142.8	Sod. sulphate (Na_2SO_4)	43.2	2.50
Potassium and K		Mag. carb....(MgCO_3)..	35.3	2.05
Sodium...(Na)....	18.7	Cal. carb....(CaCO_3)..	63.9	3.70
Magnesium..(Mg)...	10.2	Iron carb....(FeCO_3)..	1.6	.10
Calcium....(Ca)....	25.6	Alumina....(Al_2O_3)...	.9	.05
Iron.....(Fe)....	.8	Silica.....(SiO_2)....	8.8	.52
Aluminum..(Al)....	.5	Bases.....(SiO_2 +).	.8	.05
Silica.....(SiO_2)..	8.8			
Bases + Si..(SiO_2 +)	.8	Total.....	166.8	9.70
Nitrates....(NO_3)...	.8			
Chloride....(Cl)....	6.8			
Sulphate....(SO_4)...	28.9			
Carbonate..(HCO_3)..	99.9			

For industrial use it is usually of less importance, and the author deprecates the waste of time spent on elaborate calcu-

lations. The public soon learns to take results and interpret them as given. The student should, however, understand the rules of such combinations based chiefly on solubilities in the presence or absence of other salts.

At various stages of chemical knowledge, results have been reported in various ways. Just now the U. S. Geological Survey has set the fashion of reporting in ions as shown in the example given.

To the engineer and inspector a knowledge of the foregoing methods is especially desirable.

The composition of the rocks and soils affects the amount and kind of dissolved substances found in water in any given locality, and the distance from the sea affects the normal chlorine which in the interior has been found within 1 part per million when not affected by salt deposits or by *pollution*.

Some idea of the composition of the water found in different parts of the world is useful as an indication of what the engineer may expect to encounter.

The water taken by man for his use is that which is on its way to the ocean. He takes it in various concentrations from rain caught before it reaches the ground to the brines leaching out salt deposits. The kind of rock and soil through which water percolates or over which it flows modifies and controls the amount of solid matter it carries in solution as it flows on its course. The following are instances of such modification. There are regions where no good water is available. The examples given, except perhaps the Dead Sea, have all been used for industrial purposes, sea water not unfrequently so.

PERCENTAGE COMPOSITION OF SALINITY IN VARIOUS
WATERS.

TAKEN FROM U. S. G. S. BULLETIN 330.

	A	B	C	D	E	F
CO ₂	51.65	34.74	59.03	.20	trace	40.02
SO ₄	1.05	14.90	.88	7.89	.31	21.73
Cl.....	.48	6.23	.59	55.11	65.81	.64
NO ₃		1.57				
Ca.....	22.94	20.42	15.25	1.23	4.73	23.25
Mg.....	4.09	5.21	10.71	3.65	13.28	5.82
Na.....	5.14	4.92	6.68	30.64	11.65	1.81
K.....	1.75	4.65	2.24	1.09	1.85	2.04
SiO ₂	9.40	6.77	2.97		trace	4.01
Fe ₂ O ₃	1.49	.15	1.65			
Al ₂ O ₃	2.01	.44				0.58
Total per cent.....	100.00	100.00	100.00	100.00	100.00	100.00
Alkalinity, parts per million.....	195	166	144	38,789	192,150	563

- A. Mississippi River at Minneapolis.
- B. Mississippi River above New Orleans.
- C. Mille Lacs Lake.
- D. Mediterranean (mid-sea).
- E. Dead Sea — surface at north end.
- F. Virginia Hot Springs, Virginia.

TABLE I.

TABLE OF HARDNESS, SHOWING THE PARTS OF CALCIUM CARBONATE (CaCO_3) IN 1,000,000 FOR EACH TENTH OF A CUBIC CENTIMETER OF WEAK SOAP SOLUTION USED.

Using 50 cc. of the sample.

Soap Solution, cc.	0.0 cc.	0.1 cc.	0.2 cc.	0.3 cc.	0.4 cc.	0.5 cc.	0.6 cc.	0.7 cc.	0.8 cc.	0.9 cc.
0.0								0.0	1.6	3.2
1.0	4.8	6.3	7.9	9.5	11.1	12.7	14.3	15.6	16.9	18.2
2.0	19.5	20.8	22.1	23.4	24.7	26.0	27.3	28.6	29.9	31.2
3.0	32.5	33.8	35.1	36.4	37.7	39.0	40.3	41.6	42.9	44.3
4.0	45.7	47.1	48.6	50.0	51.4	52.9	54.3	55.7	57.1	58.6
5.0	60.0	61.4	62.9	64.3	65.7	67.1	68.6	70.0	71.4	72.9
6.0	74.3	75.7	77.1	78.6	80.0	81.4	82.9	84.3	85.7	87.1
7.0	88.6	90.0	91.4	92.9	94.3	95.7	97.1	98.6	100.0	101.5
8.0	103.0	104.5	106.0	107.5	109.0	110.5	112.0	113.5	115.0	116.5
9.0	118.0	119.5	121.1	122.6	124.1	125.6	127.1	128.6	130.1	131.6
10.0	133.1	134.6	136.1	137.6	139.1	140.6	142.1	143.7	145.2	146.8
11.0	148.4	150.0	151.6	153.2	154.8	156.3	157.9	159.5	161.1	162.7
12.0	164.3	165.9	167.5	169.0	170.6	172.2	173.8	175.4	177.0	178.6
13.0	180.2	181.7	183.3	184.9	186.5	188.1	189.7	191.3	192.9	194.4
14.0	196.0	197.6	199.2	200.8	202.4	204.0	205.6	207.1	208.7	210.3
15.0	211.9	213.5	215.1	216.8	218.5	220.2	221.8	223.5	225.2	226.9

TABLE II.

TABLE OF HARDNESS, SHOWING THE PARTS OF CaCO_3 IN 1,000,000 FOR EACH TENTH OF A CUBIC CENTIMETER OF WEAK SOAP SOLUTION USED.

Using 10 cc. of sample of water plus 40 cc. distilled water.

Soap Solution, cc.	0.0 cc.	0.1 cc.	0.2 cc.	0.3 cc.	0.4 cc.	0.5 cc.	0.6 cc.	0.7 cc.	0.8 cc.	0.9 cc.
0.0									8.0	16.0
1.0	24.0	31.5	39.5	47.5	55.5	63.5	71.5	78.0	84.5	91.0
2.0	97.5	104.0	110.5	117.0	123.5	130.0	136.5	143.0	149.5	156.0
3.0	162.5	169.0	175.5	182.0	188.5	195.0	201.5	208.0	214.5	221.5
4.0	228.5	235.5	243.0	250.0	257.0	264.5	271.5	278.5	285.5	293.0
5.0	300.0	307.0	314.5	321.5	328.5	335.5	343.0	350.0	357.0	364.5
6.0	371.5	378.5	385.5	393.0	400.0	407.0	414.5	421.5	428.5	435.5
7.0	443.0	450.0	457.0	464.5	471.5	478.5	485.5	493.0	500.0	507.5
8.0	515.0	522.5	530.0	537.5	545.0	552.5	560.0	567.5	575.0	582.5
9.0	590.0	597.6	605.5	613.0	620.5	628.0	635.5	643.0	650.5	658.0
10.0	665.5	673.0	680.5	688.0	695.5	703.0	710.5	718.0	726.0	734.0
11.0	742.0	750.0	758.0	766.0	774.0	781.5	789.5	797.5	805.5	813.4
12.0	821.5	829.5	837.5	845.0	853.0	861.0	869.0	877.0	885.0	893.0
13.0	901.0	908.5	916.5	924.5	932.5	940.5	948.5	956.5	964.5	972.0
14.0	980.0	988.0	996.0	1004.0	1012.0	1020.0	1028.0	1035.5	1043.5	1051.5
15.0	1059.5	1067.5	1075.5	1084.0	1092.5	1101.0	1109.0	1117.5	1126.0	1134.5

TABLE III.

TABLE OF HARDNESS, SHOWING THE PARTS OF CaCO_3 IN 1,000,000 FOR EACH TENTH OF A CUBIC CENTIMETER OF STRONG SOAP SOLUTION USED.

Using 50 cc. of sample.

Soap Solution, cc.	0.0 cc.	0.1 cc.	0.2 cc.	0.3 cc.	0.4 cc.	0.5 cc.	0.6 cc.	0.7 cc.	0.8 cc.	0.9 cc.
0.0				0.0	1.6	4.8	7.9	11.1	14.3	16.9
1.0	19.5	22.1	24.7	27.3	29.9	32.5	35.1	37.7	40.3	42.9
2.0	45.7	48.6	51.4	54.3	57.1	60.0	62.9	65.7	68.6	71.4
3.0	74.3	77.1	80.0	82.9	85.7	88.6	91.4	94.3	97.1	100.0
4.0	103.0	106.0	109.0	112.0	115.0	118.0	121.1	124.1	127.1	130.1
5.0	133.1	136.1	139.1	142.1	145.2	148.4	151.6	154.8	157.9	161.1
6.0	164.3	167.5	170.6	173.8	177.0	180.2	183.3	186.5	189.7	192.9
7.0	196.0	199.2	202.4	205.6	208.7	211.9	215.1	218.5	221.8	225.2

TABLE IV.

TABLE OF HARDNESS, SHOWING THE PARTS OF CaCO_3 IN 1,000,000 FOR EACH TENTH OF A CUBIC CENTIMETER OF STRONG SOAP SOLUTION USED.

Using 10 cc. of sample of water plus 40 cc. distilled water.

Soap Solution, cc.	0.0 cc.	0.1 cc.	0.2 cc.	0.3 cc.	0.4 cc.	0.5 cc.	0.6 cc.	0.7 cc.	0.8 cc.	0.9 cc.
0.0				0.0	8.0	24.0	39.5	55.5	71.5	84.5
1.0	97.5	110.5	123.5	136.5	149.5	162.5	175.5	188.5	201.5	214.5
2.0	228.5	243.0	257.0	271.5	285.5	300.0	314.5	328.5	343.0	357.0
3.0	371.5	385.5	400.0	414.5	428.5	443.0	451.0	471.5	485.5	500.0
4.0	515.0	530.0	545.0	560.0	575.0	590.0	605.5	620.5	635.5	650.5
5.0	665.5	680.5	695.5	710.5	726.0	742.0	758.0	774.0	789.5	805.5
6.0	821.5	837.5	853.0	869.0	885.0	901.0	916.5	932.5	948.5	964.5
7.0	980.0	996.0	1012.0	1028.0	1043.5	1059.5	1075.5	1092.5	1109.0	1126.0

TABLE V.

TABLE FOR THE PHOTOMETRIC DETERMINATION OF SULPHURIC ACID.

Depth in cm.	SO ₃ Parts per Million.	Depth in cm.	SO ₃ Parts per Million.	Depth in cm.	SO ₃ Parts per Million.	Depth in cm.	SO ₃ Parts per Million.
1.0	522.	4.0	140.	7.0	81.	10.0	57.
1.1	478.	4.1	137.	7.1	80.	10.2	56.
1.2	442.	4.2	133.	7.2	79.	10.4	55.
1.3	410.	4.3	131.	7.3	78.	10.6	54.
1.4	383.	4.4	128.	7.4	77.	10.8	53.
1.5	359.	4.5	125.	7.5	76.	11.0	52.
1.6	338.	4.6	122.	7.6	75.	11.2	51.
1.7	319.	4.7	119.	7.7	74.	11.4	50.
1.8	302.	4.8	117.	7.8	73.	11.6	49.
1.9	287.	4.9	115.	7.9	72.	11.8	48.
2.0	273.	5.0	113.	8.0	71.	12.0	47.
2.1	261.	5.1	110.	8.1	70.	12.2	47.
2.2	250.	5.2	108.	8.2	69.	12.4	46.
2.3	239.	5.3	106.	8.3	68.	12.6	45.
2.4	230.	5.4	104.	8.4	68.	12.8	44.
2.5	221.	5.5	103.	8.5	67.	13.0	43.
2.6	213.	5.6	101.	8.6	66.	13.5	42.
2.7	205.	5.7	99.	8.7	65.	14.0	41.
2.8	198.	5.8	97.	8.8	64.	14.5	39.
2.9	191.	5.9	96.	8.9	64.	15.0	38.
3.0	185.	6.0	94.	9.0	63.	15.5	37.
3.1	179.	6.1	93.	9.1	62.	16.0	36.
3.2	173.	6.2	91.	9.2	62.	16.5	35.
3.3	168.	6.3	90.	9.3	61.	17.0	34.
3.4	164.	6.4	88.	9.4	60.	17.5	33.
3.5	159.	6.5	87.	9.5	60.	18.0	32.
3.6	155.	6.6	86.	9.6	59.	18.5	31.
3.7	151.	6.7	84.	9.7	59.	19.0	30.
3.8	147.	6.8	83.	9.8	58.	19.5	29.
3.9	144.	6.9	82.	9.9	57.	20.0	29.

TABLE VI.

FOR THE CONVERSION OF PARTS PER 1,000,000 INTO GRAINS PER GALLON,
AND VICE VERSA: ALSO, FOR COMPARING DEGREES
OF HARDNESS.

Parts in 1,000,000.	Grains in U. S. Stan- dard Gallon.	Grains in Imperial Gallon.	Degrees of Hardness.		
			French: Parts CaCO ₃ in 1,000,000.	English: Grains CaCO ₃ in Imperial Gallon.	German: Parts CaO in 1,000,000.
1	.0584	.07	1	.07	0.6
2	.1167	.14	2	.14	1.1
3	.1751	.21	3	.21	1.7
4	.2335	.29	4	.28	2.2
5	.2919	.35	5	.35	2.8
6	.3502	.42	6	.42	3.4
7	.4086	.49	7	.49	3.9
8	.4670	.56	8	.56	4.5
9	.5254	.63	9	.63	5.0
17.131	1	1.1992	14.	1	08.
34.262	2	2.3893	29.	2	16.
51.393	3	3.5975	43.	3	24.
68.524	4	4.7967	67.	4	32.
85.655	5	5.9958	71.	5	40.
102.786	6	7.1950	86.	6	48.
119.917	7	8.3942	100.	7	56.
137.048	8	9.5934	114.	8	64.
154.179	9	10.7925	128.	9	72.
14.286	0.8339	1	1.8	.12	1
28.571	1.6678	2	3.6	.25	2
42.857	2.5017	3	5.4	.38	3
57.143	3.3356	4	7.1	.50	4
71.428	4.1695	5	9.0	.63	5
85.714	5.0033	6	10.7	.75	6
100.000	5.8372	7	12.6	.88	7
114.286	6.6711	8	14.3	1.00	8
128.571	7.5050	9	16.1	1.13	9

TABLE VII.

	Specific Gravity at 15° C.	Per cent Strength.
H ₂ SO ₄ (Oil of vitriol).....	1.84	100
Sulphuric acid.....	1.82	90
	1.73	80
	1.61	70
	1.39	50
HCl (Hydrochloric acid).....	1.20	40.7
	1.18	36.7
	1.12	24.4
	1.10	20.8

By weight:

	Specific Gravity at 20° C.
50 per cent alcohol.....	.914
60 per cent alcohol.....	.892
70 per cent alcohol.....	.868
80 per cent alcohol.....	.844
90 per cent alcohol.....	.818
95 per cent alcohol.....	.805

50 per cent is made from 100 cc. of 95 per cent alcohol + 90 cc. water.
 60 per cent is made from 100 cc. of 95 per cent alcohol + 70 cc. water.
 50 per cent is made from 100 cc. of 90 per cent alcohol + 84.7 cc. water.
 60 per cent is made from 100 cc. of 90 per cent alcohol + ~~15.0~~ 53.6 cc. water.

TABLE VIII.

QUANTITY OF PURE REAGENTS REQUIRED TO REMOVE ONE POUND OF
INCRUSTING OR CORROSIVE MATTER FROM THE WATER.

Incrusting of Corrosive Substance held in Solution.	Amount of Reagent. (Pure.)	Foaming Matter Increased.
Sulphuric acid.....	0.57 lb. lime plus 1.08 lb. soda ash.....	1.45 lb.
Free carbonic acid.....	1.27 lb. lime	None
Calcium carbonate.....	0.36 lb. lime	None
Calcium sulphate.....	0.78 lb. soda ash	1.04 lb.
Calcium chloride	0.96 lb. soda ash	1.05 lb.
Calcium nitrate	0.65 lb. soda ash	1.04 lb.
Magnesium carbonate.....	1.33 lb. lime	None
Magnesium sulphate.....	0.47 lb. lime plus 0.88 lb. soda ash.....	1.18 lb.
Magnesium chloride.....	0.59 lb. lime plus 1.11 lb. soda ash.....	1.22 lb.
Magnesium nitrate.....	0.38 lb. lime plus 0.72 lb. soda ash.....	1.15 lb.
Calcium carbonate.....	1.71 lb. barium hydrate	None
Magnesium carbonate.....	4.05 lb. barium hydrate	None
Magnesium sulphate.....	1.42 lb. barium hydrate	None
Calcium sulphate.....	1.26 lb. barium hydrate	None

In precipitating the calcium sulphate, there would be also precipitated 0.74 pound of calcium carbonate or 0.31 pound of magnesium carbonate, the 1.26 pounds barium hydrate performing the work of 0.41 pound of lime and 0.78 pound of soda ash; or for reacting either on magnesium or calcium sulphate, 1 pound of barium hydrate performs the work of 0.33 pound of lime plus 0.62 pound of soda ash, and the lime treatment can be correspondingly reduced.—*Report of Committee on Water Service of the American Railway Engineering and Maintenance of Way Association*, Eng. Record, April 20, 1907.

TABLE IX.

INTERNATIONAL ATOMIC WEIGHTS FOR 1905.

(From *Journal of the American Chemical Society*, Vol. XXVII, No. 1.)

	O = 16	H = 1
Aluminum.....(Al).....	27.1	26.9
Barium.....(Ba).....	137.4	136.4
Calcium.....(Ca).....	40.1	39.7
Carbon.....(C).....	12.00	11.91
Chlorine.....(Cl).....	35.45	35.18
Chromium.....(Cr).....	52.1	51.7
Hydrogen.....(H).....	1.008	1.00
Iodine.....(I).....	126.97	126.01
Iron.....(Fe).....	55.9	55.5
Lead.....(Pb).....	206.9	205.35
Magnesium.....(Mg).....	24.36	24.18
Manganese.....(Mn).....	55.0	54.6
Nitrogen.....(N).....	14.04	13.93
Oxygen.....(O).....	16.00	15.88
Phosphorus.....(P).....	31.0	30.77
Potassium.....(K).....	39.15	38.85
Silver.....(Ag).....	107.93	107.11
Sodium.....(Na).....	23.05	22.88
Sulphur.....(S).....	32.06	31.82
Zinc.....(Zn).....	65.4	64.9

SCHEME FOR TESTING BOILER SCALE.

Drop several small fragments of the boiler scale into dilute (1:2) muriatic acid.

A brisk effervescence shows that the scale is largely composed of <i>Calcium Carbonate (Limestone).</i>	After the action of the acid has <i>nearly</i> ceased, heat the acid and scale.		
	A deep yellow color and further giving off of gas indicates:— IRON CARBONATE. After the action of the acid on the scale has ceased, dilute and pour off some of the clear liquid into another test tube.		
	The residue is the <i>Silica</i> (sand) and the <i>Clayey matter</i> .	To the clear liquid add a few drops of Chloride of Barium Solution.	
		A white powder or precipitate indicates <i>Sulphates</i> .	Solution = <i>Soluble Chlorides</i> .

CONVENIENT DATA.

- 1 boiler horsepower is computed as requiring 30 pounds of water per hour.
- 1 indicated horsepower requires, in large condensing engines, about $1\frac{1}{4}$ U. S. gallons of water evaporated per hour.
- 1 indicated horsepower requires, in small non-condensing engines, frequently as much as 7 to 8 gallons of water evaporated per hour.
- 1 pound of coal will evaporate 1 gallon of water in an ordinary boiler.
- 1000 gallons of water of 143 parts per million CaCO_3 degrees hardness deposit 1.4 pound scale in a boiler.

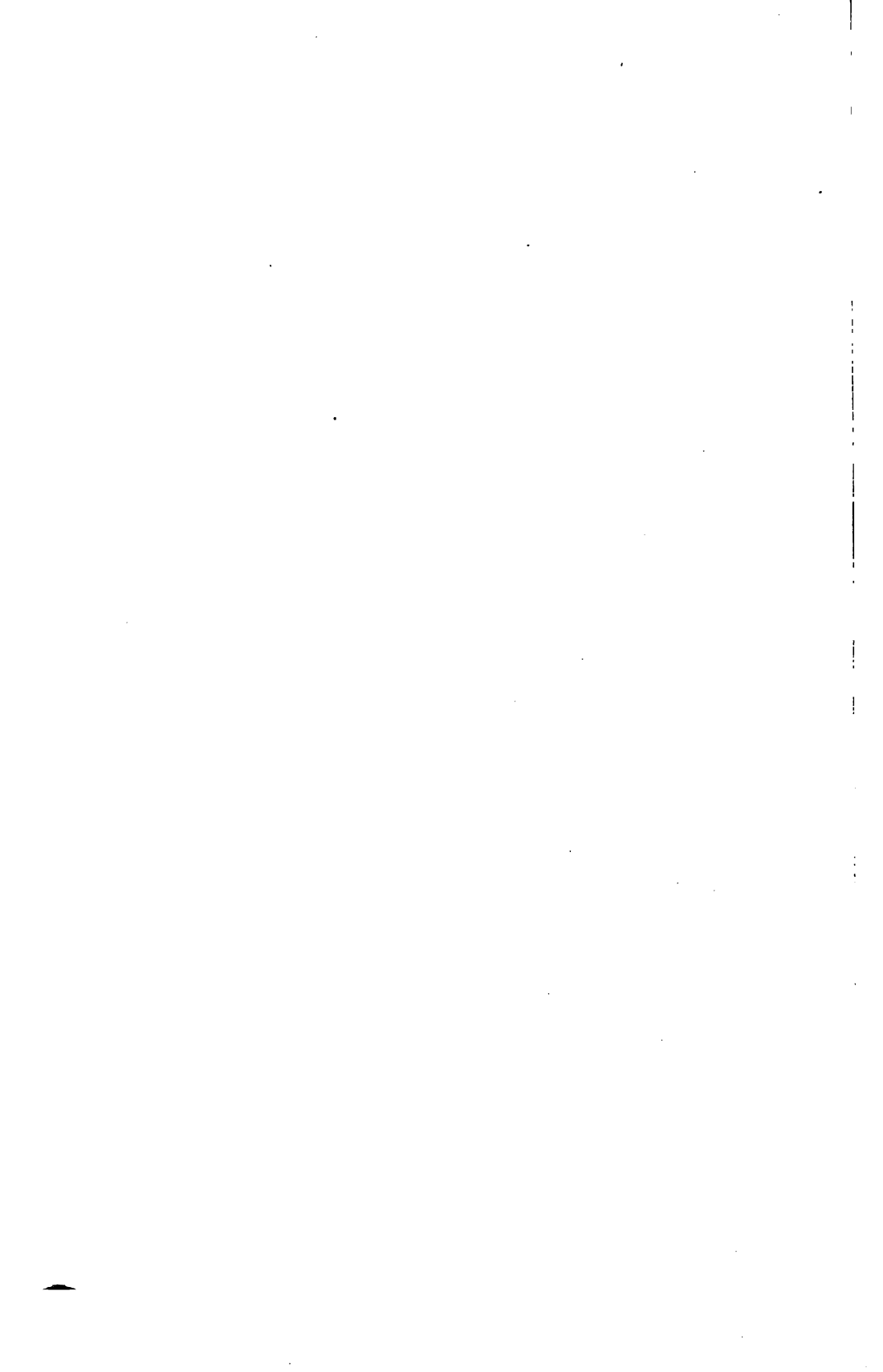
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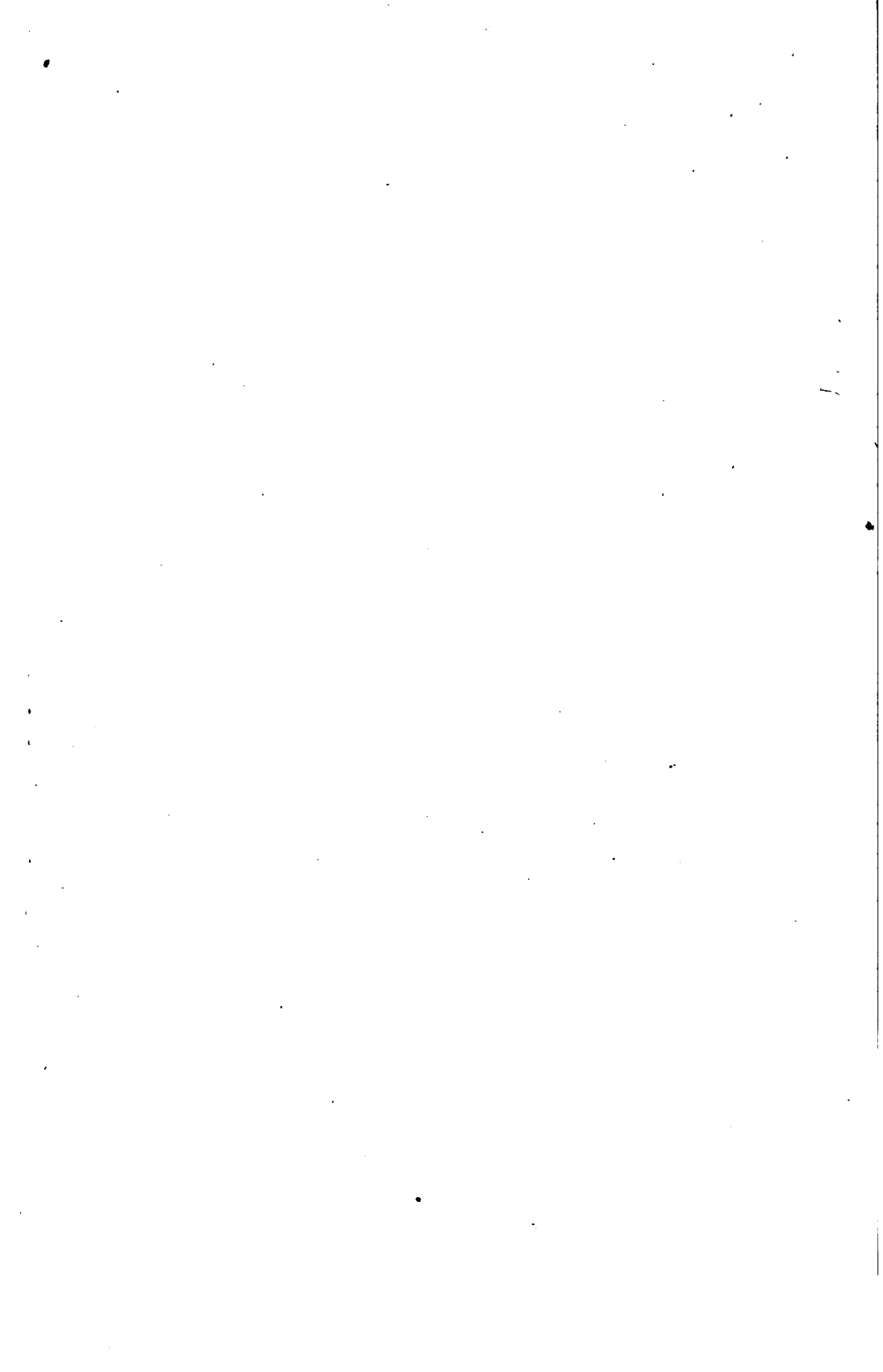
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